AFRL-RZ-WP-TP-2008-2240

POWDER METALLURGY PROCESSING OF HIGH-STRENGTH FeCo ALLOYS (PREPRINT)

Zafer Turgut, John C. Horwath, and Richard T. Fingers

Power Generation Branch
Power Division

SEPTEMBER 2008

AIR FORCE RESEARCH LABORATORY
PROPULSION DIRECTORATE
WRIGHT-PATTERSON AIR FORCE BASE, OH 45433-7251
AIR FORCE MATERIEL COMMAND
UNITED STATES AIR FORCE

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. REPORT DATE *(DD-MM-YY)*	2. REPORT TYPE	3. DATES COVERED *(From - To)*
September 2008	Technical Paper Preprint	01 October 2006 – 24 July 2008

4. TITLE AND SUBTITLE
POWDER METALLURGY PROCESSING OF HIGH-STRENGTH FeCo ALLOYS (PREPRINT)

5a. CONTRACT NUMBER
In-house

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER
62203F

6. AUTHOR(S)
Zafer Turgut (UES, Inc.)
John C. Horwath and Richard T. Fingers (AFRL/RZPG)

5d. PROJECT NUMBER
3145

5e. TASK NUMBER
01

5f. WORK UNIT NUMBER
314501CG

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
UES, Inc.
4401 Dayton-Xenia Road
Dayton, OH

Power Generation Branch (AFRL/RZPG)
Power Division
Air Force Research Laboratory, Propulsion Directorate
Wright-Patterson Air Force Base, OH 45433-7251
Air Force Materiel Command, United States Air Force

8. PERFORMING ORGANIZATION REPORT NUMBER
AFRL-RZ-WP-TP-2008-2240

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
Air Force Research Laboratory
Propulsion Directorate
Wright-Patterson Air Force Base, OH 45433-7251
Air Force Materiel Command
United States Air Force

10. SPONSORING/MONITORING AGENCY ACRONYM(S)
AFRL/RZPG

11. SPONSORING/MONITORING AGENCY REPORT NUMBER(S)
AFRL-RZ-WP-TP-2008-2240

12. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release; distribution unlimited.

13. SUPPLEMENTARY NOTES
PAO Case Number: WPAFB 08-5271; Clearance Date: 27 Aug 2008. The U.S. Government is joint author of this work and has the right to use, modify, reproduce, release, perform, display, or disclose the work. Paper contains color.

14. ABSTRACT
Fe-Co alloys are extensively used in lamination form, but there are certain power generation applications that require Fe-Co rotors in bulk form. Experiencing only a DC magnetic field, these rotors can be as large as 0.5 meter in diameter, depending on the size of the generator. The forging of such large pieces of Fe-Co has proven to be difficult. The present study investigates powder metallurgy (PM) processing of a gas atomized FeCoNbV alloy through hot isostatic pressing (HIP) for manufacturing large size rotors with improved mechanical strength. Gas atomized FeCoNbV alloy powders with and without ball milling were HIP'ed at temperatures between 675 °C and 800 °C at a fixed pressure of 193 MPa for up to 6 hours. Ball milling prior to HIP improved the yield strength. A further improvement in yield strength and in ductility was obtained after a disordering heat treatment at 730 °C followed by a rapid quench to room temperature. The optimum HIP and annealing conditions resulted in samples with yield strengths of 870 MPa. The compacts exhibited average coercivity values of 6.4 Oe and maximum permeability values of 1100.

15. SUBJECT TERMS
Iron cobalt, FeCo, soft magnetic materials

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT:	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON (Monitor)
a. REPORT	b. ABSTRACT	c. THIS PAGE			John C. Horwath
Unclassified	Unclassified	Unclassified	SAR	20	19b. TELEPHONE NUMBER *(Include Area Code)* N/A

Standard Form 298 (Rev. 8-98)
Prescribed by ANSI Std. Z39-18

Powder Metallurgy Processing of High Strength FeCo Alloys

Zafer Turgut[1], John C. Horwath[2] and Richard T. Fingers[2]

[1]UES Inc., 4401 Dayton-Xenia Rd., Dayton OH, USA

[2]AFRL, Wright-Patterson AFB, OH, USA

Abstract

Fe-Co alloys are extensively used in lamination form, but there are certain power generation applications that require Fe-Co rotors in bulk form. Experiencing only a DC magnetic field, these rotors can be as large as 0.5 meter in diameter, depending on the size of the generator. The forging of such large pieces of Fe-Co has proven to be difficult. The present study investigates powder metallurgy (PM) processing of a gas atomized FeCoNbV alloy through hot isostatic pressing (HIP) for manufacturing large size rotors with improved mechanical strength. Gas atomized FeCoNbV alloy powders with and without ball milling were HIP'ed at temperatures between 675 °C and 800 °C at a fixed pressure of 193 MPa for up to 6 hours. Ball milling prior to HIP improved the yield strength. A further improvement in yield strength and in ductility was obtained after a disordering heat treatment at 730 °C followed by a rapid quench to room temperature. The optimum HIP and annealing conditions resulted in samples with yield strengths of 870 MPa. The compacts exhibited average coercivity values of 6.4 Oe and maximum permeability values of 1100.

1. Introduction

High yield strength, high toughness, longer low cycle fatigue life, and high creep resistance are important material properties for components that are subjected to high stresses and temperatures in use, for example magnetic rotors of power generators. Mainly due to their high saturation induction and high Curie temperature, which cannot be matched by any other magnetic system, Fe-Co based alloys are the material of choice for many airborne power generators. Driven by the need for energy savings and miniaturized devices, high-power-density, lightweight and compact machine designs may be realized by further increasing rotational speeds of the power generation systems. These increased rotational speeds put extra emphasis on the strength. The mechanical properties of FeCo based alloys have been the subject of a number of studies in recent years [1-6]. There are a number of factors that influence the mechanical properties of the Fe-Co system; grain size, alloying elements, precipitates, and the final heat treatment that determines the degree of order/disorder. Influence of these parameters is well studied and documented. In the disordered state, FeCo alloys exhibit a higher strength than the ordered state. The most common alloying additive is vanadium that

suppresses the ordering, increases ductility and electrical resistivity of FeCo alloys. In another commercially available variant, Hiperco®50HS, niobium is added along with vanadium for grain refinement. Further alloying is not common, since a number of studies have demonstrated that solid solution and precipitation have little influence on strength. The incremental increase they may provide on mechanical strength deteriorates magnetic quality. Instead, the strength is mainly influenced by the grain size and materials with smaller grain size show higher yield strength (Hall-Petch relationship) as well as higher low cycle fatigue strength. A recent comprehensive review on the subject is given by Sourmail [7].

As is the case for all magnetically soft metallic systems that operate under a changing flux, FeCo alloys are used in lamination form to minimize the effect of eddy currents during operation. However, there are emerging power generation systems that utilize Fe-Co based rotors in bulk form. Experiencing only DC magnetic flux, the rotors of these generators can be as large as 0.5 meter in diameter, depending on the power output required. A rotor of this size operated at high rotational speeds requires excellent mechanical properties. Adequate fatigue and yield strength values can easily be realized in cold rolled laminations or cold rolled laminations annealed at relatively low temperatures without full recrystallization, but at the cost of inferior magnetic properties. Because this approach is based on the use of laminations, and require substantial amount of cold deformation, this practice is not suitable for producing bulk rotors. The forging of such large pieces of Fe-Co has proven to be very difficult, and perhaps impossible. A powder metallurgy (P/M) processing route using hot isostatic pressing (HIP) seems to be the only alternative for manufacturing these bulk rotors with controlled grain size, resulting in adequate strength and magnetic quality.

A common practice for P/M processing is using starting powder materials that are pre-alloyed and atomized under an inert gas or water or material powders that are milled down from a coarser powder precursor. In gas atomized powders, the atomizing gas that is in contact with liquid metal is absorbed or trapped in closed pores during processing. The same is true for vapors in liquid atomized powders. Milling under an inert gas also causes gas entrapment. If not fully eliminated, these absorbed or trapped gasses contribute to the final microstructure and product quality during P/M processing. Particle size and/or grain sizes achievable with atomization are also limited to several microns. This study focuses on milling of these atomized powders under vacuum conditions prior to consolidation for further decreasing the particulate or grain size, eliminating or minimizing defects and removing trapped or absorbed gasses in the powder, hence improving the mechanical strength of the final product. Figure 1 represents the

flow chart of the approach carried out in this study. What is uncommon in this flow chart is the vacuum ball milling which provides a significant increase in the strength of bulk compacts.

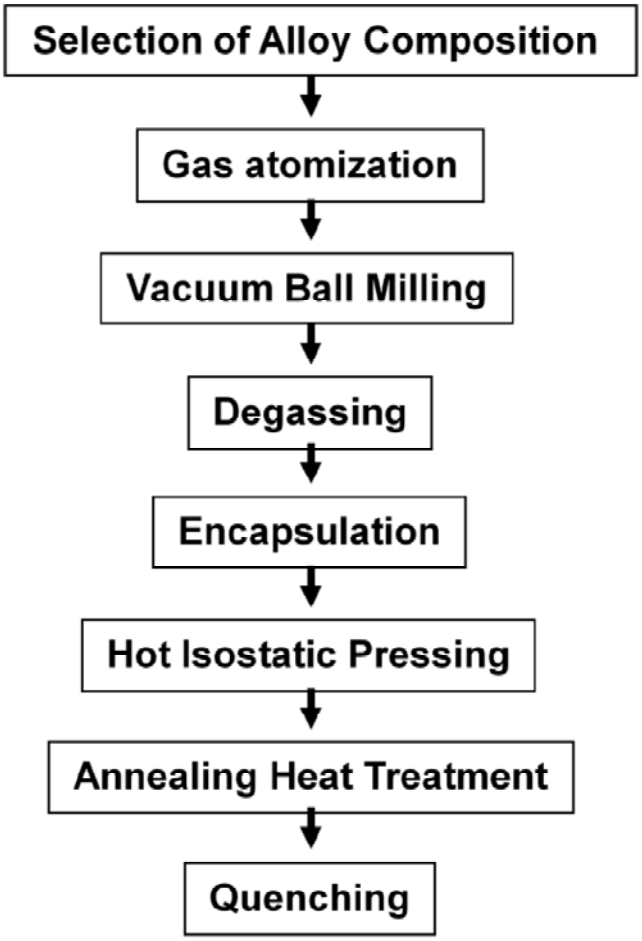


Figure 1. P/M Processing route for high strength material synthesis

2. Experimental

Three hundred kilograms of Fe-Co alloy with a nominal composition of 48.7 wt% Fe, 1.9 wt% V, 0.3 wt% Nb, balance Co was gas atomized under high purity argon gas. Gas atomized powders were then ball milled either under vacuum (~50 millitorr) or argon (~870 torr) using stainless steel balls with a powder to ball ratio of 1/20 by mass for HIP'ing studies. Ball milling times were varied between 7 and 20 hours. In order to understand the effect of vacuum milling, the evolution of gases was monitored under the conditions that mimic the heating during HIP'ing. Equal amounts of as-atomized, argon milled, vacuum milled and helium milled powders were heated up to 750 ^{o}C under vacuum and out-gassing was recorded by a gas chromatographer. The hot isostatic pressing was performed on relatively small bulk samples measuring 1.6 cm in

diameter and 10 cm. in length. After a degassing heat treatment at 730 °C for 45 minutes, vacuum milled and as-atomized powders were sealed in steel canisters and HIP'ed at temperatures between 675 °C and 850 °C at a fixed pressure of 193 MPa for up to 6 hours. A 193 MPa HIP pressure was employed because many commercial HIP vessels are limited to this pressure. After the HIP, the compacts were subjected to an ordering or disordering heat treatment in which the samples were heated up to 730 °C followed by a slow furnace cooling (ordered) or water quenching (disordered). Mechanical properties were determined by using an Instron 4505 tensile machine. Powder morphology and sizes were determined by using a JOEL JSM 6060 Scanning Electron Microscope (SEM). Microstructural characterizations were carried out using a Philips EM 420 transmission electron microscope.

3. Results and Discussion

a. Gas Atomization and Powder Milling

Gas atomization resulted in spherical powder particles with diameters anywhere from a few microns (μm) to hundreds of microns. Figure 2 shows the morphologies of the gas atomized powders. Larger particles however were agglomerates of smaller particles with an average grain size of 4-5 microns. This average grain size is very similar to that of cold rolled and fully re-crystallized laminations of the similar alloy composition (Hiperco 50 HS) and may present the lowest achievable grain size without powder milling. The powder composition after atomization was Bal. Co, 49.5 wt% Fe, 0.3 wt% Nb, 1.9 wt% V which was very close to the nominal composition with O_2 = 482 ppm and N_2 = 9 ppm.

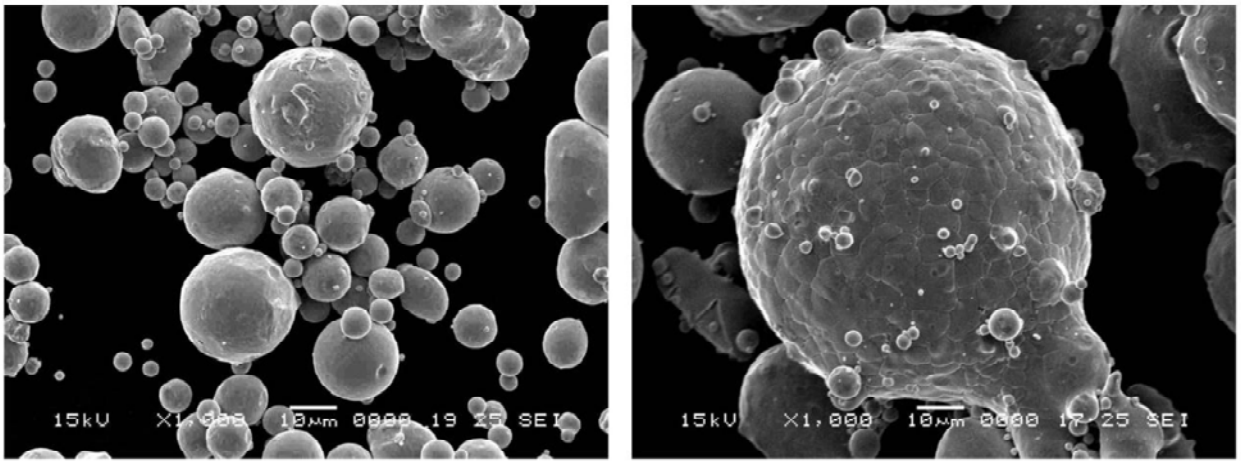


Figure 2. SEM images of gas atomized powders

Although HIP studies were only conducted on as produced and vacuum ball milled powders, powder milling was also performed under argon and helium atmospheres to study the gas entrapment during milling.

During HIP, densification occurs as a result of plastic deformation and power law creep and is a function of both applied pressure and the temperature. As densification is carried out above the yield stress of the specimen at a given temperature, considerable amount of deformation takes place by plastic flow of the particles and creep processes such as Nabarro–Herring creep (diffusion through grain interiors), Coble creep (diffusion around grain boundaries), and dislocation creep operate at relatively high rates. HIP'ing progresses through the following steps [8]:

(a) Rearrangement of particles through fragmentation of agglomerates and plastic flow.

(b) Formation of isolated pores.

(c) Collapse of the isolated pores under high external pressure.

At the last stage of the HIP'ing, if the isolated pore contains a soluble gas (O_2, N_2, H_2) it diffuses to the surface and the pore then collapses. The most common atomizing gas, argon, however is insoluble in metals and alloys. The solubility of argon in molten iron was found to be significantly lower than 1 at. ppb [9]. While this insolubility limits the gas contamination during atomization process, it prevents the collapse of the isolated pores if the argon is present in the powder as a trapped gas. The pore reduces to a diameter until the trapped argon establishes an equilibrium pressure that counteracts the external pressure preventing the pore from collapsing. One can imagine the detrimental effects of these pores on mechanical properties. Because of the mechanism described above, it is necessary to eliminate any trapped insoluble gas that may come from the atomization process or originates from the milling if it is performed under an inert gas.

Milled powders revealed two distinct morphologies (Figure 3). Some powder particles were heavily milled/deformed to a flake-like morphology with ~1 μm thickness while others were lightly deformed. One can make the argument that these lightly deformed particles have not reached a critical dislocation density which make them brittle and cause them to mill to a finer size. This will be more obvious when the microstructures of the compacts are examined. Compared to milling under an inert gas, vacuum milling significantly reduces the milling time. This may be attributed to the elimination of gas turbulence and aerodynamic drag forces under vacuum.

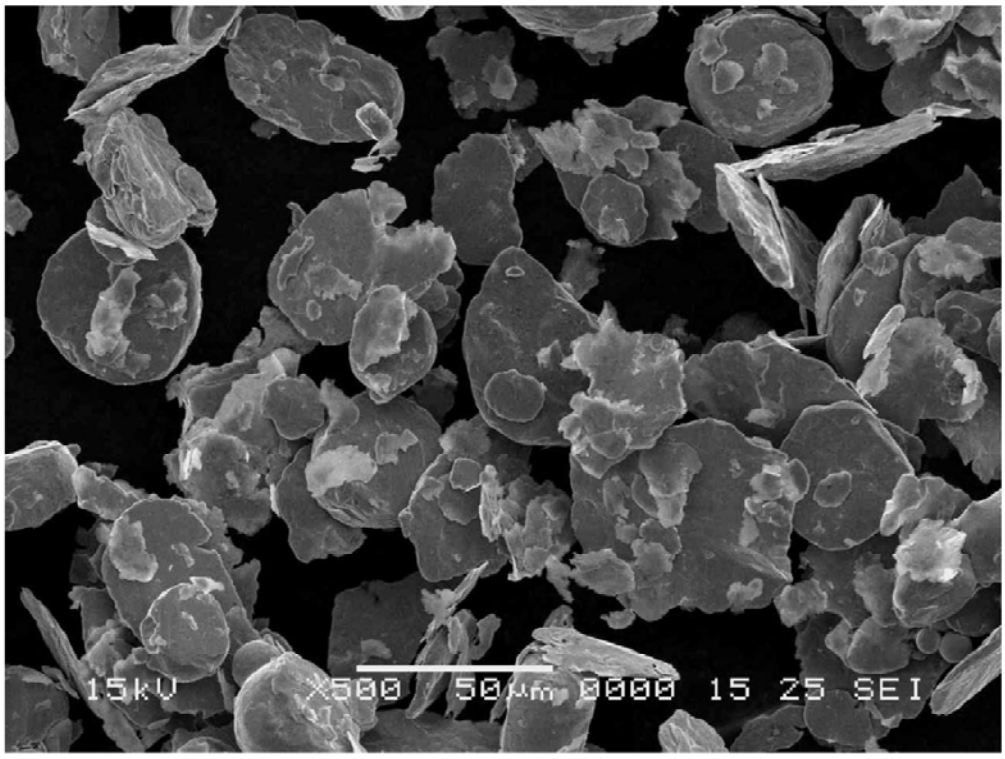


Figure 3. SEM image of vacuum ball-milled powders

b. Gas Evolution in Powders

In order to understand the influence of vacuum milling and its effectiveness to eliminate trapped gasses, the evolution of gases was monitored under the conditions that mimic heating during HIP processing. Equal amounts of as-atomized, vacuum, argon and helium milled powders were heated at a constant rate up to 750 ^{o}C under vacuum and out-gassing was recorded by a gas chromatographer. The chromatographic data in Figure 4 gives the detector response against retention time for as atomized (a), argon milled (b), and vacuum milled (c) powders. The out gassing of argon in as-atomized powders (Figure.4a) gives a clear indication that the atomizing gas, argon, is trapped in the powder particles during the atomization process. When this powder is milled under an argon atmosphere, the base line argon partial pressure increases (Figure 4b.) and a second evolution peak is observed. This secondary peak may be attributed to argon gas that is trapped during the milling process. A significant decrease in the partial pressure of argon is measured in vacuum-milled powders (Figure 4c.) confirming that vacuum ball milling is an effective method to eliminate trapped gasses. For further confirmation of gas entrapment during milling process, helium gas was also used as an inert gas medium during milling and its' evolution on heating is given in Figure 5. The evolution of helium gas in helium milled powders upon heating clearly proves this hypothesis.

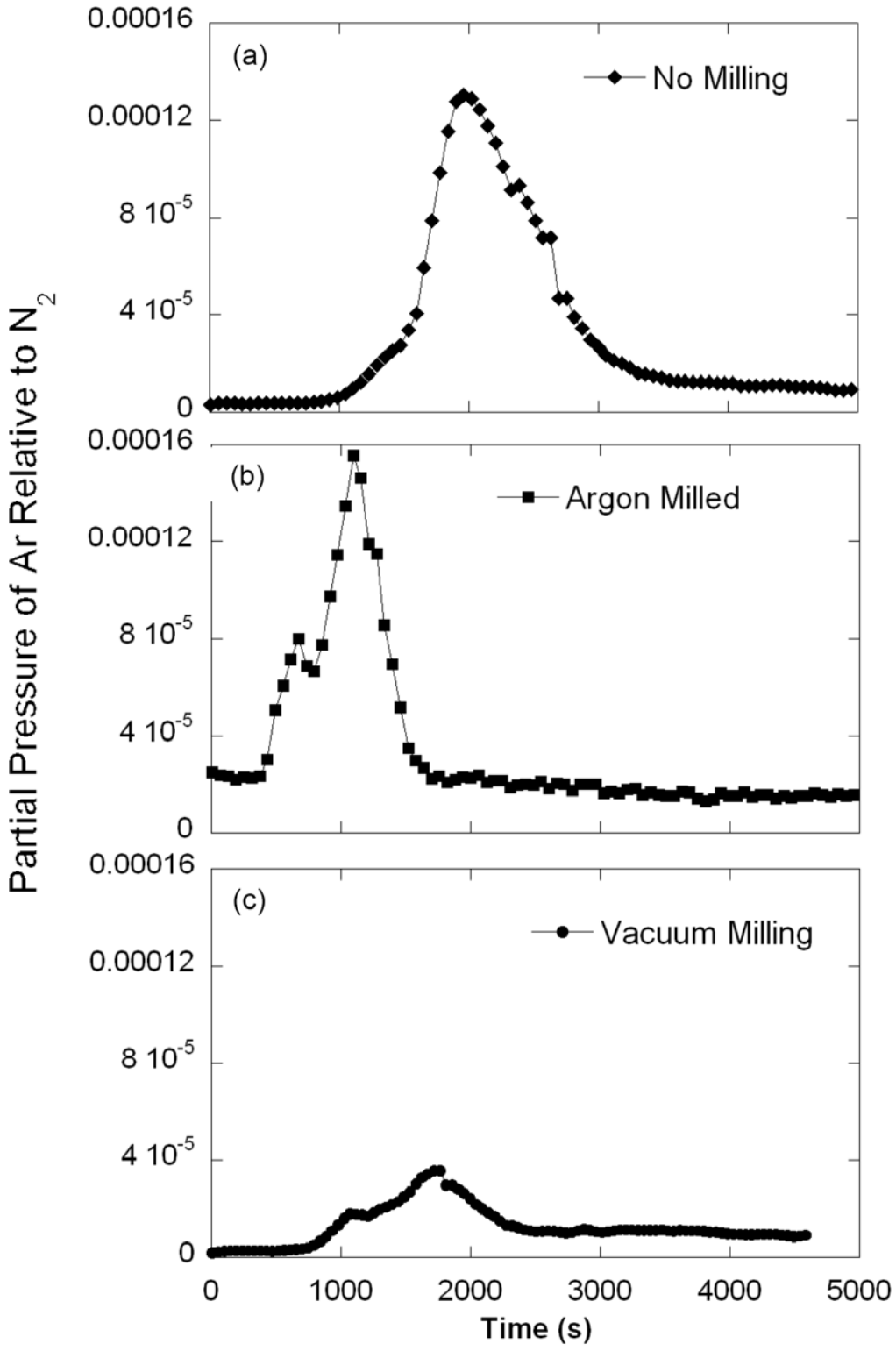


Figure 4. Detector response against retention time for as atomized (a), argon milled (b), and vacuum milled (c) powders.

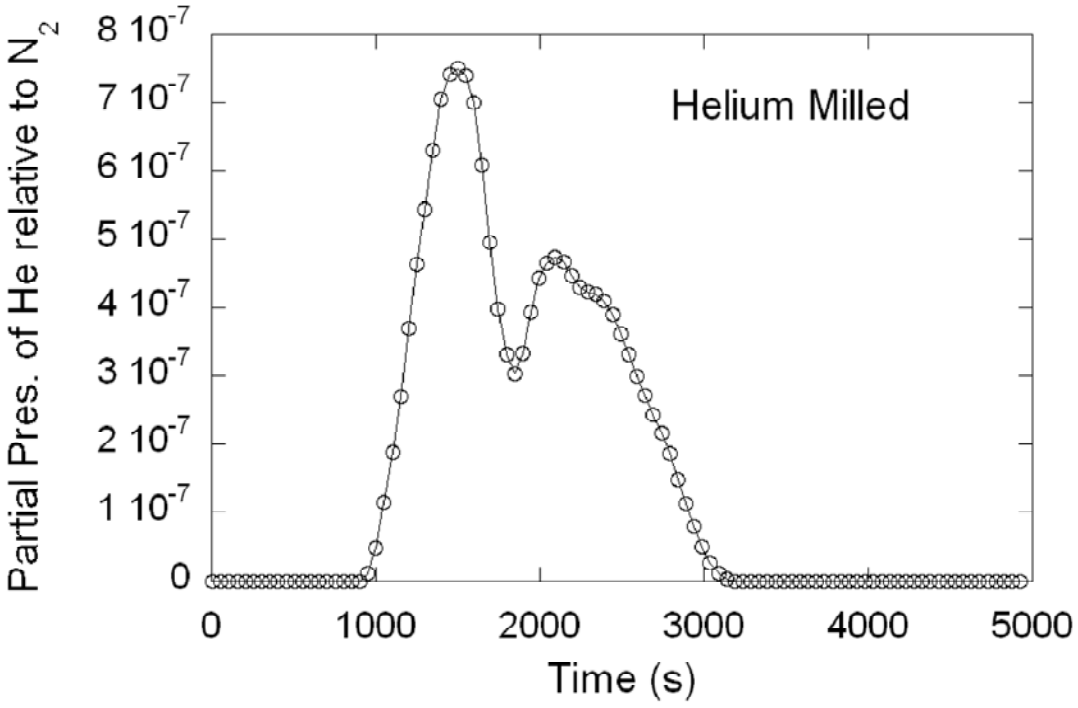


Figure 5. Detector response against retention time for helium milled powders.

c. *Hot Isostatic Pressing*

Hot isostatic pressing experiments were carried out with a fixed HIP pressure of 193 MPa while HIP temperatures were varied from 680 ^{o}C to 800 ^{o}C. HIP times also varied between 60 to 540 minutes. Regardless of the holding time, the 680 ^{o}C HIP'ed samples did not exhibit full densification. At 730 ^{o}C, the compacts were porosity free, but particle boundaries were clearly identifiable on SEM images. HIP'ing temperatures of 800 °C or higher produced full densification, but also caused a significant grain growth. The optimum consolidation temperature was found to be 750 °C. At this temperature full densification is achieved with no or minimal grain growth that resulted in yield strengths twice that of commercially available FeCo materials.

d. Yield Strength and Effect of Heat Treatment

Figure 6 shows the yield strengths of HIP'ed materials that were consolidated at 750 °C for various annealing and milling conditions. The results are given for samples that are ball milled for 20 hours under vacuum. A general trend is that yield strength increases with increasing milling time. Increasing HIP time also increases the yield strength until the full densification is reached for both milled and as-produced powders. Ball milling of the powder prior to HIP

improves the yield strength about 35 %. As HIP'ed specimens were very brittle and did not exhibit any yield during tensile testing. When ordered, samples prepared from both ball milled and as-produced powders have limited ductility and lower yield strengths than samples in the disordered state. The yield strength increase by ball-milling can be explained by the well known Hall-Petch relationship that relates the tensile yield strength to grain size.

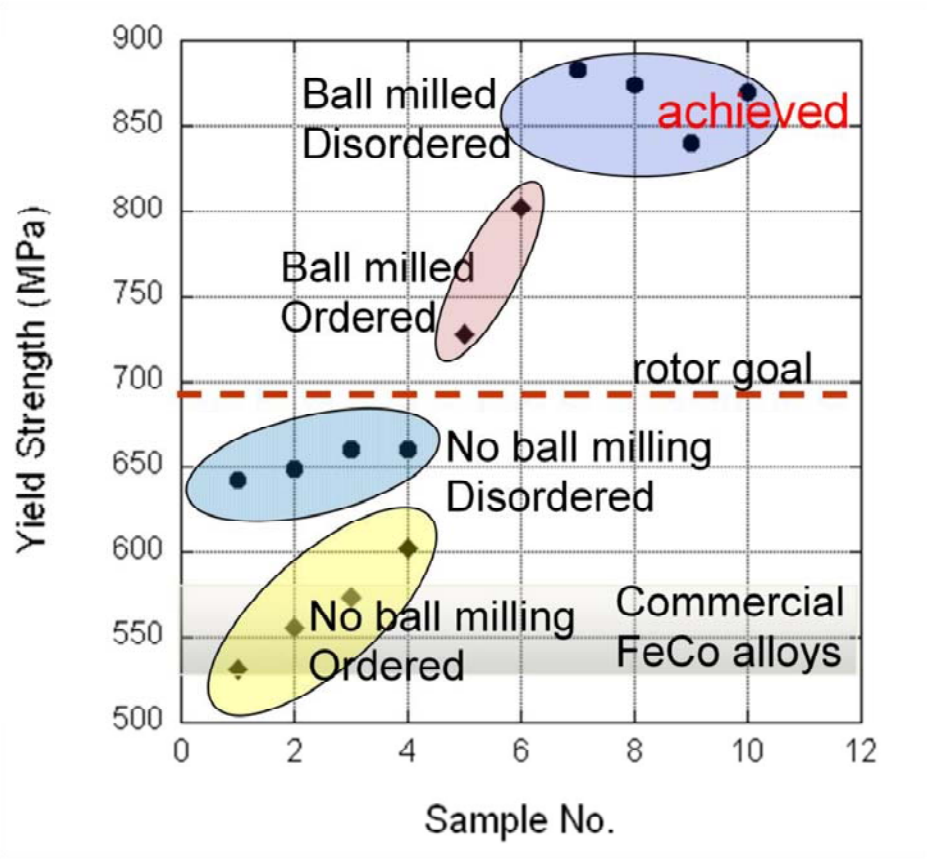


Figure 6. Yield strengths of 750 ^{o}C HIP'ed FeCoVNb materials for given annealing and milling conditions

The influence of a disordering heat treatment on the yield strength of FeCo alloys has been explored by various authors [7-10]. It was experimentally observed that the yield strength exhibits a peak for quench temperatures slightly below the order-disorder temperature. At this peak temperature deformation mode also changes from wavy to planar as the system orders. A possible mechanism proposed by Stoloff and Davies [10] suggests that slip is dominated by single dislocations in disordered systems where long range order is minimal or absent. In ordered systems where the degree of long range order is high, slip is dominated by superlattice dislocations. While superlattice dislocations are insensitive to the degree of order, motion of the single dislocations is affected by the degree of order. As the degree of order is increased, the

single dislocations experience increasing resistance. At a critical degree of order, the mechanism changes and the slip is increasingly dominated by superlattice dislocations and the strength decreases. A more detailed discussion on possible mechanisms can be found in a recent review by T. Sourmail and the references therein [7].

The highest yield strengths we obtained averaged about 870 MPa. Mechanical property data from the main US manufacturer of FeCo alloys, Carpenter Steel Corporation [11], lists the yield strength of a cold rolled and 760 °C annealed FeCo-2V alloy as 448 MPa. Although subjected to similar temperatures and longer holding times during HIP, compared to their cold-rolled and annealed counterparts, our PM processed materials exhibit almost two times higher yield strength values.

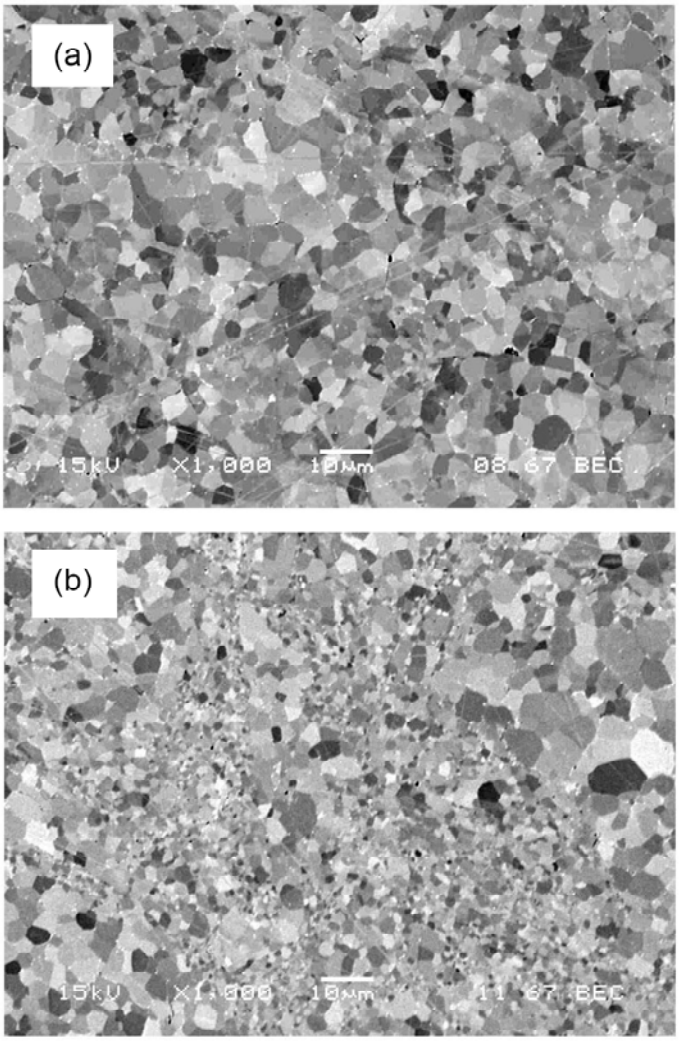
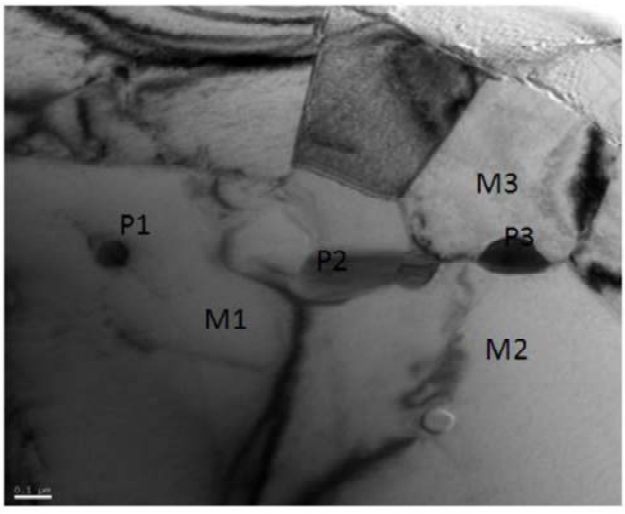


Figure 7. SEM images of HIP'ed compacts; no-milling (σ_o= 649 MPa), (a) and vacuum-milled (σ_o = 883 MPa), (b)

e. *Microstructure*

SEM images of compacts produced from gas-atomized powder revealed a uniform grain size (Figure 7a). The average grain size of ~5-7 microns indicates minimal or no grain growth during consolidation. Compacts of ball milled powders, however, exhibit two distinct regions with different grain sizes (Figure 7b). The effect of ball milling is clearly seen in the regions where the grain sizes are about 1 μm. The regions with larger grain sizes correspond to the powder particles that did not mill or deform during the milling process indicate that the ball-milling times employed are not long enough to mill/deform all the powder material.

TEM studies on ball-milled and HIP'ed compacts revealed similar Nb rich precipitates (~100 nm) with the same structure found in cold-rolled and recrystallized laminations of similar composition [12]. Figure 8 shows a bright field image of a ball-milled and HIP'ed compact along with compositional analysis by energy dispersive x-ray spectroscopy (EDS) on the matrix and the precipitates. In the figure M refers to the matrix while P refers to the precipitates. The Fe and Co content of the matrix is close to that of the nominal composition of the master alloy, but Nb is present only in precipitates along with V that is in slightly higher concentration than in the matrix. Selected area diffraction patterns (SAD) indicated that the precipitates are hexagonal Laves Phase (Figure 9) with lattice parameters a = 4.79 and c = 7.94 A^o. As expected the matrix is bcc with a = 2.855 A^o.



	Fe (at%)	Co (at%)	V (at%)	Nb (at%)
P1	28.41	49.32	2.52	19.73
P2	20.73	45.71	2.03	31.50
P3	36.05	49.02	1.93	12.99
M1	48.46	49.66	1.87	0.00
M2	49.74	48.59	1.65	0.00
M3	49.16	49.27	1.55	0.00

Figure 8. Bright field image and compositional analysis (EDS) of 750 ^oC HIP'ed compact

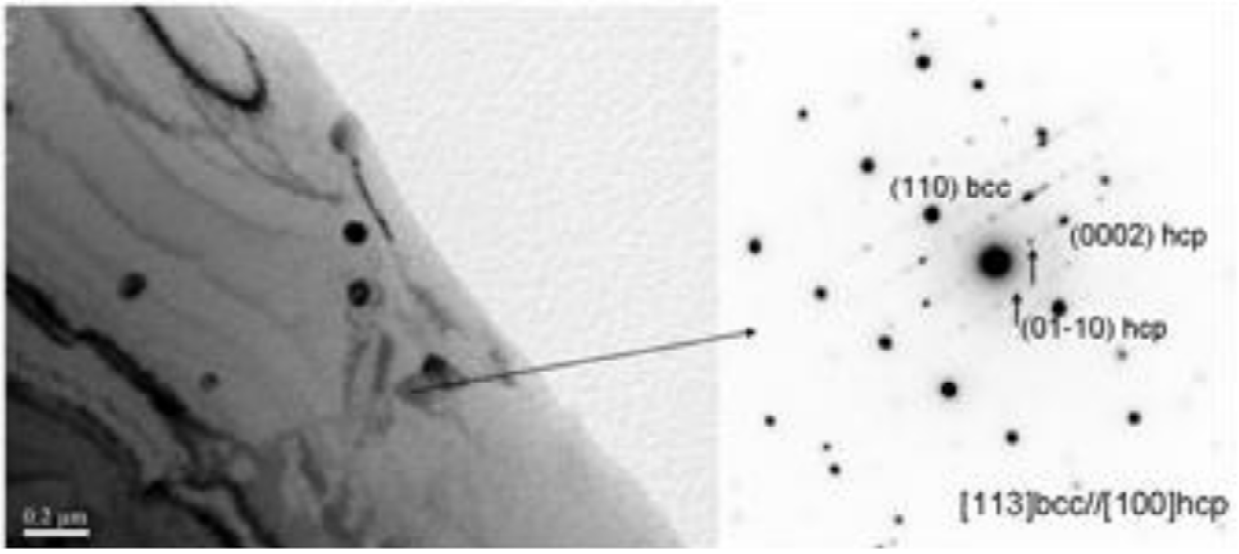


Figure 9. Bright field image and SAD pattern of a precipitate

f. Magnetic Properties

Since this study is intended for manufacturing a rotor that operates under DC flux conditions, magnetic property requirements for such a rotor are relatively relaxed. Other than having the highest possible saturation flux density, such rotor designs generally call for a magnetic material with coercivity about 20 Oe or lower and full saturation under applied magnetic fields of 200 Oe or less. Our compacted samples exceeded these requirements, with average coercivity values of 6.4 Oe and maximum permeability values of 1100. Considering the fact that the ball-milling times employed in this study were not long enough to completely mill/deform all the powder material to a greater extend, it should be possible to improve the yield strength of these alloys beyond the values obtained in this study, while still meeting the magnetic property requirements.

4. Acknowledgements

The authors would like to thank Jack Coate and John Murry of AFRL for tensile testing and HIP'ing respectively.

5. References

1. A. Duckham, D.Z. Zhang, D. Liang, V. Luzin, R.C. Cammarata, R.L. Leheny, C.L. Chien, and T.P. Weihs, *Acta Mater*. **51** (2003) 4083

2. R.S. Sundar, S.C. Deevi, *Intermetallics* **12** (2004) 92

3. E. Hug, O. Hubert, I. Guillot, *J. Magn. Magn. Mater.* **215,216** (2000) 197

4. L. Ren, S. Basu, R.-Hai Yu, J. Q. Xiao, A. Parvizi-Majidi, *J. Mat. Sci*. **36** (2001) 1451

5. E.P. George, A.N. Gubbi, I. Baker, L. Robertson, *Mater. Sci. and Eng*. **A329–331** (2002) 325

6. R. H. Yu, S. Basu, L. Ren, Y. Zhang, A. Parvizi-Majidi, K. M. Unruh, and J. Q. Xiao, *IEEE Trans. Magn.*, **36**, (2000) 3388

7. T. Sourmail, *Prog. Mater. Sci.* **50** (2005) 816

8. H.V. Atkinson, S. Davies, *Met. Mat. Trans.,* **31A**, (2000) 2981

9. K. Ishii, Y. Sasaki, K. Tokuchi, Y. Kashiwaya, *Metal. Mater. Trans. B* **31B** (2000) 216

10. N.S. Stoloff, R.G. Davies, *Acta. Metall*. **12** (1964) 473

11. Carpenter Steel Corporation Material Data Sheet

12. J.C. Horwath Z. Turgut and R.T Fingers, High Temperature Properties and Aging Stress Related Changes of FeCo Materials. *Technical Report AFRL-PR-WP-TR2006-2176 Air Force Research Laboratory, Wright-Patterson Air Force Base, Ohio*; (2006)